DRAFT

Comments on behalf of the Responsible Party on the

Comment [pb1]:

Draft - M/V *Selendang Ayu*
2008 Study Plan for Assessment of Remaining Oil

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Summary

The trustees proposed study is apparently intended to provide the information ~~necessary~~that will assist in ~~for plann~~planning restoration activities for the area of northern Unalaska Island affected by the December 8, 2004 *Selendang Ayu* oil spill in compliance with Natural Resource Damage Assessment Regulations.

However, ~~T~~the Draft Study Plan, as written, is not clearly related to assessing injuries, if any, to natural resources. Though the Plan focuses on "~~-~~shoreline segments of greatest concern", and this "focus will be on a series of shoreline segments with a high likelihood of remaining oil and/or those with biological concerns evidenced by previous study results", these specific biological concerns are not stated. As a result, rather than representing a plan that is focused on specific biological resources (e.g., harlequin ducks) the plan consists of a collection of measurements from several types of samples. Of great concern is the fact that the methods of analysis of the resultant data from these samples are questionable and are disconnected from the potential biological concerns (e.g., harlequin duck exposures). A practical, focused, and reasonable plan would be one that consisted of measurements of the bioavailability of any remaining oil and hence risk to wildlife through invertebrate prey species that could be ingested by wildlife. This should be the emphasis of the Plan.

In order for the Plan to represent a cooperative and focused effort, the RP and the trustees need to agree on study sites, sample types, and especially on how the data will be analyzed and used (data analysis methods, relevant criteria/endpoints against which the data will be compared). These latter facets are left out of the Plan. They need to be based on accepted practice rather than experimental research performed in this study. Significant disagreements may result if such data interpretation criteria or endpoints are not fully vetted and agreed upon to avoid potentially differing conclusions.

The ~~use of reference sites have~~use of reference sites has been included in the Plan as proposed. The Plan's use of the term "minimally oiled sites" is confusing and possibly misleading. If the sediments contain spilled oil at any level then they are not "reference sites'. If they contain no such oil, then they should be called "reference sites".

Recommendations on use of data and identification benchmarks/criteria/endpoints are included in this summary. Specific recommendations include:

1. Reliance on bioavailability data from invertebrates rather than PEMDs
2. Careful conversion of any PEMD data to approximate (i.e., order of magnitude) corresponding water quality data so as to facilitate comparisons to widely acceptable water quality criteria
3. Comparison of any sediment PAH data to NOAA-developed sediment quality guidance (i.e., the ER-L and ER-M values)
4. An increase in the number of sediment samples that are analyzed for chemistry data so as to permit a true representation of the oil remaining on the beaches
5. Careful evaluation of location of any remaining oil with regards to tide zonation and assessment of risk to corresponding biota from the tide levels
6. Elimination of the inappropriate use of the "W" parameter as a sole basis for determining "toxicity potential" of sediments

The Draft Study Plan defines the environmental chemistry program that will be used by the Trustees to evaluate the presence, weathering state and potential toxicity of residual oil that may be present on beaches within the *M/V Selendang Ayu* spill zone. Given the complexity of the spill (e.g., mixtures of two different oils), possible presence of secondary sources, and availability of relevant analytical data from other field programs, recommendations have been provided to ensure that the quality of the data is sufficient to complement the stated study objectives and that the chemical data will be compatible with previous studies performed at the site. Additional specific recommendations include:

1. Laboratory Workplan preparation with specific Data Quality Objectives that are compatible with the prior environmental chemistry work at the site.

2. Development/application of an oil mixing model combined with Nordtest source ratio analysis to confirm the identity of the oil.

3. Direct measurements of oil weathering using C_{30} 17α(H),21β(H)-hopane based percent oil depletion estimates.

Study Components

The study as written has three objectives:

1. *Determine the presence, distribution, and relative amount of oil remaining on beach segments of greatest concern to determine if Selendang Ayu oil remains on shorelines within the core spill area.*

2. *Determine the weathering state of remaining oil to evaluate the potential toxicity of remaining oil.*

3. *Determine bioavailability of the remaining oil to assist in evaluating exposure and potential biological effects.*

~~Comments on each of these areas follows~~Comments on each of these areas follow.

Shoreline Sediments. Concerning #1 above, the search for remaining oil on the beaches, the study as planned seeks to locate any remaining oil irrespective of tide zone and therefore independent of any biological resources. It would be reasonable to select resources (e.g., intertidal biota) and focus on sediments that are associated with these resources. Instead, it appears that all tide zones, including the supratidal, where oil from the *M/V Selendang* had previously been observed, is being sampled. This is unnecessary and not useful. (Note: measures of the bioavailability of this oil are relevant and are covered in #3). Data analysis methods should be included. How are the data on the pits sampled to be used? What extrapolations of individual pit data to the entire segment, if any, will be made and how will such extrapolations be justified?

It appears that visual (and olfactory) evidence of oil will be relied on heavily to identify the distribution of subsurface oil in pits dug on the shore. According to Table 5, approximately 795 pits will be dug in 10 oiled segments (including harlequin duck sampling sites and subsistence sampling sites). An estimated total of 217 sediment samples will be collected from the 795 pits (0.27 samples per pit or about one sample for every four pits). According to the text, these samples may include surface oiling samples, so the number of sediment samples per pit will be less than 0.25 samples per pit. According to the text, "Up to 60 sediment samples will be analyzed, to represent different zones and the range of visual oiling conditions." This represents less than 30 % of the sediment samples collected. There is a large variation in the total oil (TPH) or total polycyclic aromatic hydrocarbon (PAH) present in subsurface sediments in a given visual oil category (e.g., HOR, MOR, LOR). Presumably, the 60 samples will be used to develop a correlation between visual oiling categories and total PAH concentration. This sampling and analysis effort will be insufficient to accurately estimate the "relative amount" of oil remaining on the shore.

Chemical Composition and Weathering. The Plan discusses the chemical analyses of the sediments. The Plan seems to disconnect the issue of bioavailability from the issue of oil remaining in sediments. Any inference of the possible exposure to and toxicity of any remaining oil without demonstrating bioavailability of any buried residues seems illogical. Thus it seems that a data analysis sequence asking the question "is the remaining oil bioavailable (#3, above) should be answered first.

The trustees state (page 12) that, "Sampling of surface and/or subsurface oil (we presume they mean "sediment") is intended to evaluate the weathering state of remaining oil within each identified oiled zone." If this is the sole objective of the sediment sampling/analysis program, objective 1 will not be addressed adequately. The weathering state of the Selendang Ayu oils (heavy fuel and diesel) and the computation of the weathering parameter "W", as determined by a correlation model developed by Short and Heintz (1997) for estimating weathering of the North

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Slope crude oil spilled from the *Exxon Valdez*, ~~can not~~cannot be used to estimate the weathering state and toxicity of the residues of Selendang Ayu fuel oils. The weathering model was based on the PAH composition of the North Slope crude oil and weathering patterns observed in oiled sediments in Prince William Sound. Because the physical properties and composition of the oils spilled from the Selendang Ayu and the coastal sediments on Unalaska Island where some of the oil accumulated are quite different from the composition of the oil spilled from the *Exxon Valdez* and the coastal sediments in Prince William Sound and the western Gulf of Alaska where some of the oil accumulated, it is unlikely that the weathering model of Short and Heintz (1997) will give reliable estimates of weathering state of Selendang Ayu oils, particularly of more highly weathered samples that are poorly characterized by the model.

The model depends heavily on changes in specific PAH analyte ratios in the oil as it weathers. Ratios of most parent and alkyl PAH are different in the fresh Selendang Ayu oil from those in fresh North Slope crude oil and effects of weathering on changes in these ratios are likely to be different because of differences in physical properties of the oils and composition of other components (especially heavy ends) of the two oils. Thus, the Short and Heintz (1998) weathering model must be validated independently with Selendang Ayu oil before it can be used to estimate weathering state of the Selendang Ayu oil.

The Study Plan states, *"the toxic potential of PAH in sediment will be estimated by comparing observed weathering condition in PEMD and sediment samples to weathering known to cause significant damage to fish"*. There is simply no scientifically defensible way to use "W" in sediment samples to say anything about possible toxicity, without taking into account the concentration of key toxicants. "W" values alone and especially those derived using a model developed for another oil (i.e., North Slope Crude Oil) can not used for the purpose stated. As indicated in Table 6, there is no correlation between *w* and the toxicity of North Slope crude oil. We also question the validity of using the oil residues in PEMDs to estimate *w*. As Carls et al. (2004) point out, the PAH composition in the PEMD following deployment is different from the PAH composition in tissues of marine animals (e.g., salmon embryos) concurrently exposed to the oil. PEMDs accumulate and retain higher molecular weight PAH to a greater extent than low molecular weight 2-ring PAH. The rate of partitioning of PAH from oil decreases as the oil weathers, because of PAH depletion from the oil phase and increasing viscosity of the oil, decreasing the bioavailability and toxicity of the oil. All these rate processes are likely to be different for heavy fuel oil and North Slope crude oil; thus, a weathering parameter, based on laboratory observations of weathering of North Slope crude oil, can not be used to predict the toxicity of heavy fuel oil.

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In summary, "W", a measure of weathering (or composition of the remaining oil) has been applied to *water* exposures to fish in laboratory studies (e.g., Carls et al., 1999, 2005). "W" has never been applied to field *sediment* concentrations; the cited publications refer to exposures of fish, larvae, and eggs through the *water* in experimental settings. PAH *concentrations* in water (not sediments) have been used in other spills by the trustees as indicators of toxicity. While the "W" parameter does give a measure of the degree of weathering compared to the spilled oil itself, "W" has never been used as a standalone parameter for the determination of oil toxicity. "W" is a dimensionless parameter and does not indicate the concentration of oil in the sediments or the concentration of the PAH compounds. Toxicity cannot and should not be inferred from the

"W" parameter. Toxicity can only be inferred from a combination of total PAH concentration and those of key compounds (e.g., naphthalenes). Thus, as proposed, the use of "W" in this Plan is not acceptable.

A more defensible *criterion* (i.e., a well-used surrogate for toxicity) for the evaluation of sediment data is the NOAA sediment quality limits or guideline approach of Long and Morgan. These guidelines set the ER-L (lowest, most conservative estimate of the PAH levels at which any effects are first noticed) at about 4000 ppb PAH and the ER-M (the median concentration at which adverse effects are likely to occur) at about 47,000 ppb PAH. Neither is a direct measure of toxicity, but instead are well-used (and accepted) guidelines (Note: the ER-L is the most conservative of the PAH sediment guidelines. The RP and the trustees should agree on whether the ER-L or the ER-M will be used and then how any exceedance of either will be interpreted.)

Bioavailability. Two measures of bioavailability are proposed – intertidal invertebrates (mussels, etc.); and passive samplers (SPMDs), specifically in this Plan, PEMDs (i.e., plastic strips). (Note: PEMDs are not SPMDs, in that the inner olein layer built into SPMDs, which were designed to simulate the affinity of hydrocarbons for lipids within organisms, is missing in the PEMDs). These two methods – biota (e.g., mussels) and PEMDs - cannot be considered of equal merit for two reasons. Simply stated, ducks and other wildlife eat intertidal biota; they do not eat PEMDs. Analysis of biota taken from the sites is by far the more relevant and applicable measures of bioavailability. Such samples should be the main focus. In the absence of indigenous biota at any of the shoreline segments to be studied, caged mussels should be deployed.

Mussels are integrated measures of bioavailable hydrocarbons (including PAH) representing exposures over time. They take up hydrocarbons from the water, equilibrate, and depurate. On the other hand, any passive samples, including the proposed PEMDs, do not depurate; thus whatever is adsorbed on the PEMD surface is an overestimate of what truly is bioavailable to ducks and other wildlife.

Among the choices of the various passive samplers (i.e., SPMDs) those that involve the uptake of dissolved hydrocarbons through a membrane and into olein within the membrane best mimic the sample process of equilibration across a biological membrane and into the lipids of the animal. The PEMDs, as developed by Carls et al (2004), does not include the olein layer. It merely includes a plastic strip upon which hydrocarbons (dissolved, particulate) may be irreversibly adsorbed. Thus while they are certainly easy to use and are convenient, the problem is in the interpretation of the results.

Hydrocarbons adsorbed on the PEMD plastic cannot be equated to hydrocarbons that may be taken up by animals. In addition, it is not at all clear what a concentration of adsorbed hydrocarbons on the PEMD plastic strip equates to in terms of the analogous concentration in water. It is this *water* concentration that should be the key data product of any investigation. Mussel concentration can easily be converted to water concentrations and there are many publications on this conversion (e.g., Neff and Burns, 1996). Not so for the PEMDs.

Neither the use of PEMDs of the evaluation of the "w" parameter provides a prediction of the dose to which intertidal animals, particularly the valued ecosystem components (e.g., harlequin

ducks), will be exposed. Finally, we do not know the critical dose (the dose causing ecologically significant biological effects) to valued ecosystem components of the bioavailable fractions (PAH and biodegradation products, etc.) of the heavy fuel oil that has weathered to different degrees (different *w* values) in intertidal sediments. Therefore, we can not predict injury to fish and wildlife (a critical component of NRDA) based on measures of oil weathering and bioavailability.

With regards to the proposed PEMD deployments themselves, PEMDs deployed offshore with care are likely to be free of any artifact when retrieved. However, PEMDs deployed in mesh bags on top of the sediment are highly likely to be fouled and/or contaminated with sediment after 28 days as the sediment mixes and moves on the shoreline as a result of wave action. The PEMDs may actually become buried in the sediment thus rendering them contaminated. Because of these highly likely problems, the use of PEMDs anchored in these high-energy sediments is problematic. At a minimum the trustees and the RP should agree to discard the results from any PEMD that is either a) buried during the 28-day deployment periods, or b) the mesh bag filled with fine sediments and hence "fouled".

Of further concern is the trustees' proposed data analysis method for the PEMDs. The Plan states:

> *"All of the minimally oiled PEMD samples will be used to determine the average total PAH loading. The total PAH loading on the PEMD samples from the oiled zones will be compared with the results from the minimally oiled sites. If a sample from the oiled zones is higher than the mean plus one standard deviation of the minimally oiled sites, then the answer to the question "Is the oil bioavailable?" will be yes for that sample.*

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Given the very low levels of sorbed hydrocarbons expected on the PEMDs, an adequate number of *replicates* of any samples (PEMDs or biota) need to be compared with the reference site *replicates*. The method of comparing the concentrations of PAH in single samples to the mean of the reference samples is not a rigorous comparison.

Beyond the statistical comparison method used, if PEMDs are to be deployed (in spite of the weaknesses described above), the RP and the trustees must decide on criteria to be used to interpret the data. That is, what level of PAH on any PEMD equates to an equivalent water concentration that exceeds a water quality criteria or other agreed upon criteria?

Analytical Chemistry

This section evaluates the design of the Trustee environmental chemistry program to produce defensible hydrocarbon chemistry results to support the stated study objectives and provides specific recommendations to support the proposed chemistry program.

Oil composition analysis of sediment and tissue will be used to achieve the program objectives. Therefore the analytical chemistry data must be robust enough to resolve and identify oil

source(s) and to document the degree of oil weathering in the field samples. In addition, data comparability with previous field studies will optimize the interpretive power of the proposed analytical program. To achieve these goals, a Laboratory Work Plan accompanied by rigorous Data Quality Objectives (DQO) should be prepared.

Analytical Chemistry Recommendations

1. A laboratory workplan or standard operating procedure (SOP) that defines each step of the chemical analysis should be included in the Study Plan.
2. A listing of DQO's and target analyte lists should also be clearly defined. Table 1 is a listing of the DQO's that were used by NewFields for the M/V *Selendang Ayu* field programs. Adoption of these~~/~~ or similar DQOs would insure that the chemical data produced during the proposed field program would be compatible with historical site data. The NewFields target analyte lists and associated reporting limits are also provided in Tables 2 and 3 and 4.

Source Identification

The keystone of any oil spill ~~investigation~~study is the defensible identification of the spilled oil. The plan states, *"The source of hydrocarbons in sediments and mussels will be inferred using an algorithm that summarizes three independent oil recognition models and two pyrogenic recognition models (Carls, 2006).* The authors also state that *"Each of the oil recognition models has two outputs, a generic recognition of petroleum and specific identification of Alaska North Slope (ANS) crude oil,; the combined scores range from 0-6. Previous experience with Selendang Ayu oil indicates that the model will work well for this oil (Carls 2007)[1]."*

The application of this model at this site is not warranted because 1) Identification of the presence or absence of oil in the samples will be determined by the analysis of the pyrogenic/petrogenic polycyclic aromatic hydrocarbon distributions[2], 2) The method has been calibrated with ANS crude oil and not IFO 380 fuel oil (spilled oils). 3) The spilled oil is a variable mixture of two separate *M/V Selendang Ayu* IFO 380 fuels with completely different chemical properties (Figure 1). Mixtures of these fuels were observed at the spill site, which exhibited a wide range of PAH, distributions (Figure 2) that were very different from the source oils. Reliable identification of the spilled oil with the source fuels can only be performed by the application of source specific mixing model. Given that the proposed nonparametric oil identification model (Carls 2006)[3] is not calibrated for this site, and specifically for variable mixtures of IFO fuels, it will not provide the resolution required to separate spilled oils from other possible sources.

In addition, the use of PEMD results for source identification purposes is not recommended due to analytical bias introduced during transport in the water column.

[1] Carls, M.G., Hudson, J., Rice, D.S. 2007. Selendang Ayu oil risk to early life stage salmon. Juneau, AK, NOAA/NMFS, Auke Bay Laboratory.
[2] Sauer, T., Boehm, P.D. 1991. The use of defensible analytical chemical measurements for oil spill natural resource damage assessment. Proceedings 1991 Oil Spill Conference. American petroleum Institute.
[3] Carls, M. 2006. Nonparametric identification of petrogenic and pyrogenic hydrocarbons in aquatic ecosystems. *Environ. Sci. Technology*. 2006. **40**: pp. 4233-4239.

Source Identification Recommendations**:**

1. The two primary M/V *Selendang Ayu* source oils should be analyzed with the field samples.
2. Quantitative analysis of triterpanes and steranes should be performed for source confirmation (Table 4).
3. A mixing model based on sulfur differences in the source oils should be developed and applied at each site to quantify the degree of oil mixing.
4. Nordtest[4] source ratios (Table 5) should be used to evaluate the results of the mixing model and confirm the identity of the oil.

Degree of Weathering

Since the "W' parameter and the model upon which it is based was developed for an entirely different oil (i.e., North Slope Crude Oil) a better way of evaluating weathering would be the use of a direct estimation of oil degradation or percent oil depletion from the chemical data using the methods of Douglas et al., 2002[5], Prince et al., 1994[6], or Butler et al., 1994[7].

Degree of Weathering Recommendations:

~~1.~~ A more accurate weathering index will be achieved if the field chemistry data and the source oil information are used to directly calculate the percent depletion for total oil and target alkane and PAH compounds.

[4] Daling, P.S., Faksness, L, Hansen, A.B., Stout, S.A. 2002. Improved and standardized methodology for oil spill fingerprinting. *Environmental Forensics*, **3**: pp. 263-278.

[5]Douglas, G.S., Hardenstine, J., Owens, E.H., and Prince, R.C. 2002. The OSSA II pipeline oil spill: the character and weathering of the spilled oil. *Spill Science & Technology Bulletin* **7**(3-4): pp. 135-148.

[6] Prince, R.C., Elmendorf, D.L., Lute, J.R., Hsu, C.S., Haith, C.E., Senius, J.D., Dechert, G.J., Douglas, G.S. and Butler, E.L. 1994. 17a (H) 21 ß (H)-hopane as a conserved internal marker for estimating the biodegradation of crude oil. *Environ. Sci. Technol.* 28(1): pp. 142-145.

[7] Butler, E.L., Douglas, G.S., Steinhauer, W.G., Prince, R.C., Aczel, T., Hsu, C.S., Bronson, M.T., Clark, J.R. and Lindstrom. J.E. 1991. Hopane, a new chemical tool for measuring oil biodegradation. In: R.E. Hinchee and R.F. Olfenbuttel (eds.), On-Site Bioreclamation: Processes for Xenobiotic Hydrocarbon Treatment. Stoneham, Mass.: Butterworth-Heineman.
pp. 515-521.

Figure 1. PAH distributions of the two primary fuel oils released from the *M/V Selendang Ayu*.

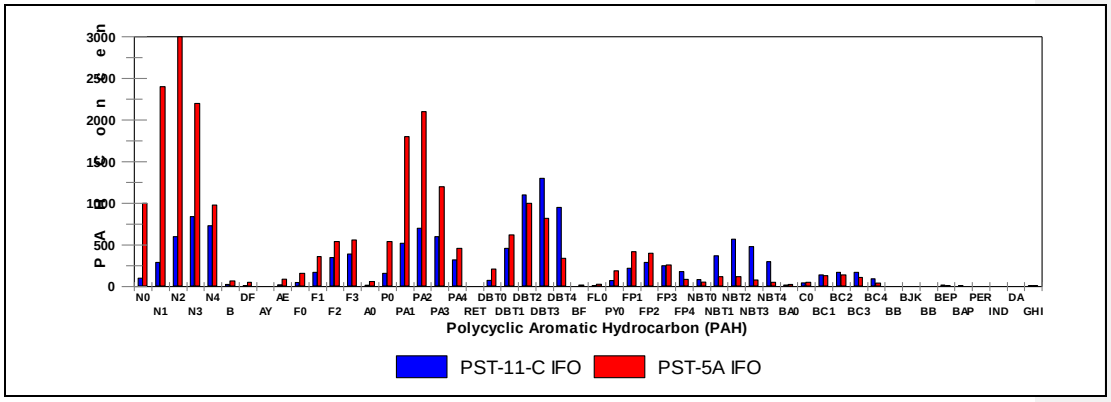

Figure 2. PAH distribution in ~~a beached~~beached oil sample. Note that the PAH distribution does not match the two source oils and is approximately a 50/50 mixture of the two source oils.

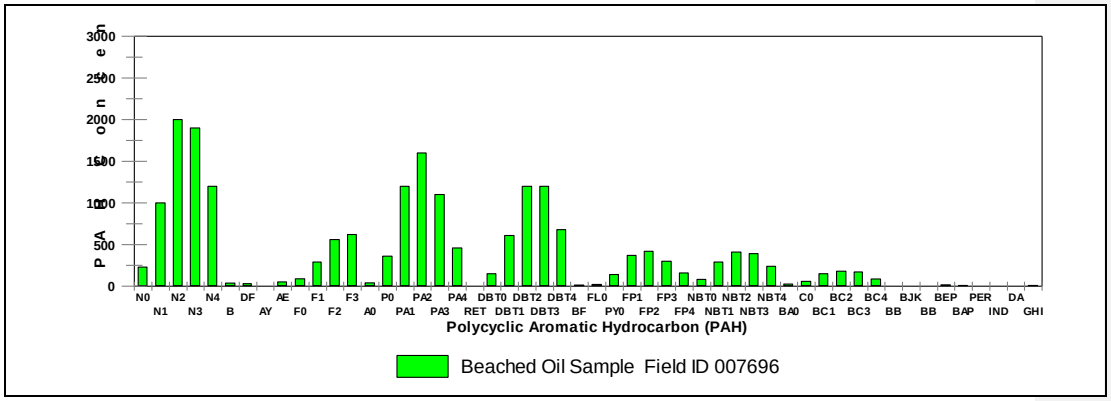

Table 1. Recommended Data Quality Objectives (DQO) for the proposed 2008 field ~~program .~~program.

Element or Sample Type	Minimum Frequency	Data Quality Objective/Acceptance Criteria	Corrective Action
MS Tuning	Prior to each run sequence using PFTBA	m/e 69: Base Peak (~100,000 counts minimum) m/e 219: 30-60% Base Peak abundance m/e 502: 5-11% Base Peak abundance	Perform Instrument Maintenance. Re-tune
MS Tune	Prior to each run sequence using DFTPP	Per "Maximum Sensitivity" Tune criteria (WHG SOP O-010) for PCBs; Per SW846 8270C for other SVOCs	Perform Instrument Maintenance. Re-tune
Initial Calibration	Prior to every instrument batch sequence or as needed indicated by continuing calibration check	5 point curve, minimum of 4 point. %RSD ≤ 25% for 90% of analytes and ≤35% for all analytes >C6. 5 point curve, minimum of 4 point. %RSD ≤ 25% for 90% of analytes. No more than 10% >35% for 8260.	Perform Instrument Maintenance. Re-calibrate. Evaluate impact to data, discuss with manager, determine if corrective action is necessary
Continuing Calibration (CCV)	Must end analytical sequence and every 12 field samples or 24 hours, whichever is more frequent	%RSD ≤25% for 90% of analytes. %RSD ≤ 35% for all analytes >C6. %RSD ≤25% for 90% of analytes. No more than 10% >35% for 8260.	Perform Instrument Maintenance. Re-analyze affected samples. Notify project manager and justify.
Procedural Blank	Every batch / every 15-20 samples	Less than the reporting limit unless analyte not detected in associated sample(s) or associated sample analyte concentration is > 5x blank value.	Re-extract samples. Evaluate impact to data, discuss with manager, determine if corrective action is necessary
Laboratory Control Sample (LCS)	Every batch/every 15-20 samples	50%- 130% recovery VOCs for 90% of the spiked analytes greater than C6 50% - 130% recovery SVOCs	Evaluate impact to data, discuss with manager, determine if corrective action is necessary
Matrix Spike (MS)	Every batch/every 15-20 samples for soil and sediment samples	50% - 150% recovery for 90% of the analytes spiked >5 times background	Evaluate impact to data, discuss with manager, determine if corrective action is necessary
Duplicate (Dup)	Second aliquot of field sample	< 30% RPD for those analytes 5 times the reporting limit	Evaluate impact to data, discuss with manager, determine if corrective action is necessary
Recovery/Surrogate Standards	Every Sample	50% - 130% recovery for other SVOCs. 70% - 130% recovery VOCs	Re-extract samples. Evaluate impact to data, discuss with manager, determine if corrective action is necessary
Internal Standard (IS)	Every Sample	50% - 200% of the area of the IS in the associated calibration standard	Evaluate impact to data, discuss with manager, determine if corrective action is necessary
Instrumental Check	One per initial calibration	80% - 120% recovery SVOC only	Perform Instrument Maintenance. Re-calibrate. Evaluate impact to data, discuss with manager, determine if corrective action is necessary
North Slope Crude	Semi VOA Only One per initial calibration PAH	65% - 135% recovery PAHs	Evaluate impact to data, discuss with manager, determine if corrective action is necessary

Table 2. TPH and Alkane Target Analyte List

Abbr.	Analyte	RL	Abbr.	Analyte	RL
nC9	n-Nonane	250 ug/Kg Dry	nC27	n-Heptacosane	250 ug/Kg Dry
nC10	n-Decane	250 ug/Kg Dry	nC28	n-Octacosane	250 ug/Kg Dry
nC11	n-Undecane	250 ug/Kg Dry	nC29	n-Nonacosane	250 ug/Kg Dry
nC12	n-Dodecane	250 ug/Kg Dry	nC30	n-Triacontane	250 ug/Kg Dry
nC13	n-Tridecane	250 ug/Kg Dry	nC31	n-Hentriacontane	250 ug/Kg Dry
1380	2,6,10 Trimethyldodecane	250 ug/Kg Dry	nC32	n-Dotriacontane	250 ug/Kg Dry
nC14	n-Tetradecane	250 ug/Kg Dry	nC33	n-Tritriacontane	250 ug/Kg Dry
1470	2,6,10 Trimethyltridecane	250 ug/Kg Dry	nC34	n-Tetratriacontane	250 ug/Kg Dry
nC15	n-Pentadecane	250 ug/Kg Dry	nC35	n-Pentatriacontane	250 ug/Kg Dry
nC16	n-Hexadecane	250 ug/Kg Dry	nC36	n-Hexatriacontane	250 ug/Kg Dry
nPr	Norpristane	250 ug/Kg Dry	nC37	n-Heptatriacontane	250 ug/Kg Dry
nC17	n-Heptadecane	250 ug/Kg Dry	nC38	n-Octatriacontane	250 ug/Kg Dry
Pr	Pristane	250 ug/Kg Dry	nC39	n-Nonatriacontane	250 ug/Kg Dry
nC18	n-Octadecane	250 ug/Kg Dry	nC40	n-Tetracontane	250 ug/Kg Dry
Pr	Pristane	250 ug/Kg Dry			
nC19	n-Nonadecane	250 ug/Kg Dry	TEH	TEH ·(C_9-C_{44})	8.25 mg/Kg Dry
nC20	n-Eicosane	250 ug/Kg Dry			
nC21	n-Heneicosane	250 ug/Kg Dry		Surrogates	
nC22	n-Docosane	250 ug/Kg Dry	OTP	O-Terphenyl	
nC23	n-Tricosane	250 ug/Kg Dry	D50T	Tetracosane-D50	
nC24	n-Tetracosane	250 ug/Kg Dry			
nC25	n-Pentacosane	250 ug/Kg Dry		Internal Standard	
nC26	n-Hexacosane	250 ug/Kg Dry	5AA	5-α-Androstane	

Table 3: PAH Target Analyte List

Compound	RL	Compound	RL
Naphthalene	2.5 ug/Kg	Benz(a)anthracene	2.5 ug/Kg
C1-Naphthalenes	2.5 ug/Kg	Chrysene	2.5 ug/Kg
C2-Naphthalenes	2.5 ug/Kg	C1-Benz(a)anthracenes/Chrysenes	2.5 ug/Kg
C3-Naphthalenes	2.5 ug/Kg	C2-Benz(a)anthracenes/Chrysenes	2.5 ug/Kg
C4-Naphthalenes	2.5 ug/Kg	C3-Benz(a)anthracenes/Chrysenes	2.5 ug/Kg
Acenaphthylene	2.5 ug/Kg	C4-Benz(a)anthracenes/Chrysenes	2.5 ug/Kg
Acenaphthene	2.5 ug/Kg	C0-Benzonaphthothiophene	2.5 ug/Kg
Biphenyl	2.5 ug/Kg	C1-Benzonaphthothiophene	2.5 ug/Kg
Dibenzofuran	2.5 ug/Kg	C2-Benzonaphthothiophene	2.5 ug/Kg
Fluorene	2.5 ug/Kg	C3-Benzonaphthothiophene	2.5 ug/Kg
C1-Fluorenes	2.5 ug/Kg	C4-Benzonaphthothiophene	2.5 ug/Kg
C2-Fluorenes	2.5 ug/Kg	Benzo(b)fluoranthene	2.5 ug/Kg
C3-Fluorenes	2.5 ug/Kg	Benzo(j/k)fluoranthene	2.5 ug/Kg
Anthracene	2.5 ug/Kg	Benzo(a)fluoranthene	2.5 ug/Kg
Phenanthrene	2.5 ug/Kg	Benzo(e)pyrene	2.5 ug/Kg
C1-Phenanthrenes/Anthracenes	2.5 ug/Kg	Benzo(a)pyrene	2.5 ug/Kg
C2-Phenanthrenes/Anthracenes	2.5 ug/Kg	Indeno(1,2,3-cd)pyrene	2.5 ug/Kg
C3-Phenanthrenes/Anthracenes	2.5 ug/Kg	Dibenz(a,h)anthracene	2.5 ug/Kg
C4-Phenanthrenes/Anthracenes	2.5 ug/Kg	Benzo(g,h,i)perylene	2.5 ug/Kg
Dibenzothiophene	2.5 ug/Kg	Retene	2.5 ug/Kg
C1-Dibenzothiophenes	2.5 ug/Kg	Perylene	2.5 ug/Kg
C2-Dibenzothiophenes	2.5 ug/Kg		
C3-Dibenzothiophenes	2.5 ug/Kg		
C4-Dibenzothiophenes	2.5 ug/Kg	Surrogates	
Benzo(b)fluorene	2.5 ug/Kg	d10-2-Methylnaphthalene	
Fluoranthene	2.5 ug/Kg	d10-Pyrene	
Pyrene	2.5 ug/Kg	d12-Benz(b)Fluoranthene	
C1-Fluoranthenes/Pyrenes	2.5 ug/Kg		
C2-Fluoranthenes/Pyrenes	2.5 ug/Kg	Recovery Internal Standard	
C3-Fluoranthenes/Pyrenes	2.5 ug/Kg	d10-Acenaphthene	
		d12-Chrysene	

Table 4. Biomarker Target Analyte List

Compound	Compound
C23 Tricyclic Terpane (T4)	13b(H),17a(H)-20S-Diacholestane (S4)
C24 Tricyclic Terpane (T5)	13b(H),17a(H)-20R-Diacholestane (S5)
C25 Tricyclic Terpane (T6)	13b,17a-20S-Methyldiacholestane (S8)
C24 Tetracyclic Terpane (T6a)	14a(H),17a(H)-20S-Cholestane (S12)
C26 Tricyclic Terpane-22S (T6b)	14a(H),17a(H)-20R-Cholestane (S17)
C26 Tricyclic Terpane-22R (T6c)	13b,17a-20R-Ethyldiacholestane (S18)
C28 Tricyclic Terpane-22S (T7)	13a,17b-20S-Ethyldiacholestane (S19)
C28 Tricyclic Terpane-22R (T8)	14a,17a-20S-Methylcholestane (S20)
C29 Tricyclic Terpane-22S (T9)	14a,17a-20R-Methylcholestane (S24)
C29 Tricyclic Terpane-22R (T10)	14a(H),17a(H)-20S-Ethylcholestane (S25)
18a-22,29,30-Trisnorneohopane-TS (T11)	14a(H),17a(H)-20R-Ethylcholestane (S28)
C30 Tricyclic Terpane-22S (T11a)	14b(H),17b(H)-20R-Cholestane (S14)
C30 Tricyclic Terpane-22R (T11b)	14b(H),17b(H)-20S-Cholestane (S15)
17a(H)-22,29,30-Trisnorhopane-TM (T12)	14b,17b-20R-Methylcholestane (S22)
17a/b,21b/a 28,30-Bisnorhopane (T14a)	14b,17b-20S-Methylcholestane (S23)
17a(H),21b(H)-25-Norhopane (T14b)	14b(H),17b(H)-20R-Ethylcholestane (S26)
30-Norhopane (T15)	14b(H),17b(H)-20S-Ethylcholestane (S27)
18a(H)-30-Norneohopane-C29Ts (T16)	C26,20R- +C27,20S- triaromatic steroid
17a(H)-Diahopane (X)	C28,20S-triaromatic steroid
30-Normoretane (T17)	C27,20R-triaromatic steroid
18a(H)&18b(H)-Oleananes (T18)	C28,20R-triaromatic steroid
Hopane (T19)	
Moretane (T20)	<u>Surrogates</u>
30-Homohopane-22S (T21)	5β(H)cholane
30-Homohopane-22R (T22)	
30,31-Bishomohopane-22S (T26)	<u>Internal Standard</u>
30,31-Bishomohopane-22R (T27)	d_{12}-Chrysene
30,31-Trishomohopane-22S (T30)	
30,31-Trishomohopane-22R (T31)	
Tetrakishomohopane-22S (T32)	
Tetrakishomohopane-22R (T33)	
Pentakishomohopane-22S (T34)	

Table 5. Nordtest Diagnostic Source Ratios Used To Identify M/V *Selendang Ayu* Spilled Oil.

#	Source Ratio
1	Nor/Pr
2	Pr/Ph
3	CPI
4	DBT/P
5	D2/P2
6	D3/D3
7	C28+C29 tri/H
8	C28+C29 tri/Ts+Tm
9	Ts/H
10	Moretane/H
11	25NH/Hop
12	BNH + 25NH/H
13	NH/H
14	% [C31 H (S/S+R)]
15	% [C32 H (S/S+R)]
16	% [C35 H / ∑C30-C35 H]